



TITLE:

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Formation of periodic steps with a unit-cell height on 6H-SiC (0001) surface by HCl etching

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Step bunching on 6H-SiC (0001)-vicinal face etched by HCl at 1300–1500 °C is investigated by atomic force microscopy. When the substrate has the inclination toward near $\langle 01\bar{1}0 \rangle$ or even $\langle 11\bar{2}0 \rangle$, continuous parallel and periodic microsteps with six-bilayer height are laid perpendicular to the off direction, although those perpendicular to $\langle 11\bar{2}0 \rangle$ are apt to decompose into three bilayer or less. Formation mechanism of unit-cell-height steps is discussed based on consideration of bond configuration at step edges. © 2000 American Institute of Physics. [S0003-6951(00)01623-5]

Surface pretreatment is an important technique to obtain superior epilayers. Especially for silicon carbide (SiC), the hardness and high chemical stability bring difficulty in removing surface polishing damage at a moderate temperature, resulting in many surface scratches observable even on commercially available wafers. On the other hand, the bunching of atomic steps on 6H- and 4H-SiC {0001} faces has been of interest, since their particular crystal structures often lead to wide terraces between microsteps with specific heights and to other particular features including interlacing.¹ Etching of 6H- or 4H-SiC (0001)-vicinal faces by H₂ at high temperatures has been reported to bunch atomic steps into microsteps with the height of each unit cell as well as to remove scratches.^{2–4} The mechanism of the step bunching was suggested to be the minimization of surface free energy. Similarly, etching by HCl was reported to have a strong impact on the surface morphology of the subsequent SiC homoepitaxial layer⁵ in chemical vapor deposition (CVD); H₂ carrier gas etches carbon in SiC leaving excess silicon, which is expected to be taken away by HCl. However, a precise study on HCl-etched surfaces has not been carried out yet. In this letter, the authors report step bunching on 6H-SiC (0001)-vicinal faces etched by HCl, and discuss the mechanism of microstep formation.

The samples used were 6H-SiC (0001)-vicinal faces specified as “on-axis” from three suppliers. The crystal orientation was determined from cleavage perpendicular to the $\langle 01\bar{1}0 \rangle$ direction and/or x-ray diffraction. The samples were etched by HCl (3 sccm) with H₂ carrier gas (1 slm) in a horizontal cold-wall CVD reactor,⁶ typically at 1300 °C for 10 min. Then, the step structure was observed by an atomic force microscope (AFM). The angle of off-axis was also estimated from the AFM observation. With the crystal orienta-

tion determined as described above, the direction of off-axis could be also estimated.

The microsteps showed different behaviors according to the off direction. In our experiment, three off directions were observed: $\langle 01\bar{1}0 \rangle$, $\langle 11\bar{2}0 \rangle$, and about 15° from both $\langle 01\bar{1}0 \rangle$ and $\langle 11\bar{2}0 \rangle$.

Figure 1 shows the AFM image of an etched surface with the off direction toward $\langle 01\bar{1}0 \rangle$. The etching was done at 1300 °C for 10 min. The microsteps are perpendicular to the $\langle 01\bar{1}0 \rangle$ direction (or, aligned along the $\langle 11\bar{2}0 \rangle$ direction) and nearly parallel and periodic almost all over the sample. The height of these microsteps is about 1.5 nm, corresponding to the height of 6H-SiC unit cell (six bilayers). The terrace width is about 0.3 μm. From these results, the off angle is estimated to be 0.3° toward $\langle 01\bar{1}0 \rangle$. Etching at 1500 °C showed similar results.

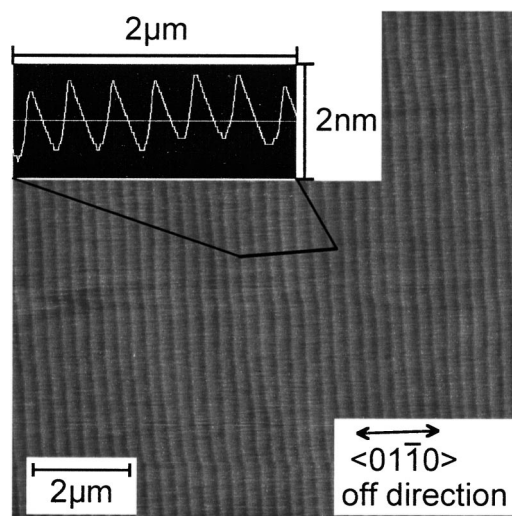


FIG. 1. AFM image of the etched surface with off direction toward $\langle 01\bar{1}0 \rangle$. The etching was done at 1300 °C. The off angle was estimated to 0.3°.

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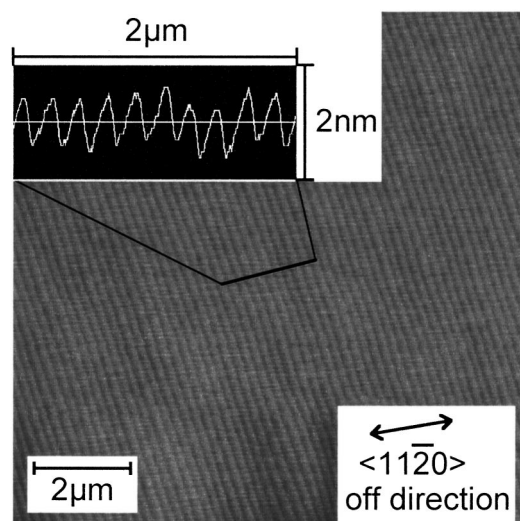


FIG. 2. AFM image of the etched surfaces with off direction toward $\langle 11\bar{2}0 \rangle$. The etching was done at 1300 °C. The off angle was estimated to 0.5°. In this case, regular microsteps were obtained.

Figures 2 and 3 show some results for the off direction toward $\langle 11\bar{2}0 \rangle$. The microsteps on the surface in Fig. 2 are perpendicular to the $\langle 11\bar{2}0 \rangle$ direction (or, aligned along the $\langle 01\bar{1}0 \rangle$ direction) with six-bilayer height, and nearly parallel and periodic. The terrace width is about 0.2 μm , and the off angle is estimated to be 0.5° toward $\langle 11\bar{2}0 \rangle$. The microsteps on some samples were apt to decompose into steps with three-bilayer height or less, which was enhanced when etched at 1500 °C. On some samples, microsteps with “triangular depressions” at step edges were observed as shown in Fig. 3(a). The “depressions” are three bilayer in height

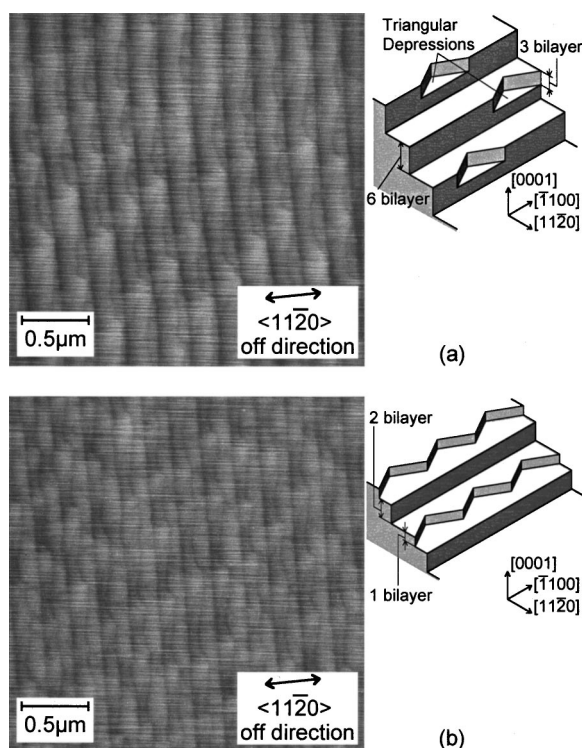


FIG. 3. AFM images of the etched surfaces with off direction toward $\langle 11\bar{2}0 \rangle$. (a) Microsteps with triangular depressions (etched at 1300 °C.) (b) Microsteps decomposed into two-bilayer-height straight steps and one-bilayer-height zigzag steps (etched at 1500 °C.)

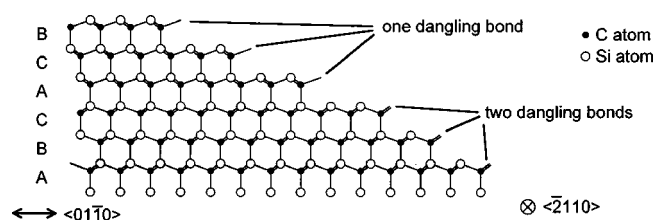


FIG. 4. Atomic bonds near the $\langle 01\bar{1}0 \rangle$ -off surface viewed from the $\langle \bar{2}110 \rangle$ direction. Larger and smaller circles stand for Si and C atoms, respectively.

and surrounded by steps perpendicular to $\langle 01\bar{1}0 \rangle$. When the microsteps completely decomposed by the etching at 1500 °C, two kinds of steps emerged alternately as shown in Fig. 3(b): straight steps perpendicular to $[11\bar{2}0]$ with two bilayer height and zigzag steps perpendicular to $[01\bar{1}0]$ or to $[10\bar{1}0]$ with one bilayer height. To the authors' knowledge, this alternation has not been reported for H_2 etching. Thus, with the off direction toward $\langle 11\bar{2}0 \rangle$, inhomogeneity of step structure was observed in different samples or even in one sample. The cause of this inhomogeneity is not clear yet, although maybe attributed to the variation of surface quality such as polishing damage.

On the sample with the off direction of about 15° from both $\langle 01\bar{1}0 \rangle$ and $\langle 11\bar{2}0 \rangle$ in the (0001) basal plane, microsteps were not continuous: in some narrow band regions the microsteps seemed to be parallel, periodic, and perpendicular to the $\langle 01\bar{1}0 \rangle$ direction, although in other regions the atomic steps seemed not to bunch. These results suggest that the step-bunching behavior should strongly depend on the off direction.

It is of scientific interest that parallel and periodic microsteps with a unit-cell height were obtained for the samples inclined toward $\langle 01\bar{1}0 \rangle$. In contrast, with the off-axis toward $\langle 11\bar{2}0 \rangle$, steps alternately emerged apparently perpendicular to $\langle 11\bar{2}0 \rangle$ and to $\langle 01\bar{1}0 \rangle$ family. In epitaxial growth, it is reported that when tilted by a small angle toward $\langle 01\bar{1}0 \rangle$, the surface shows many stripe-like undulations after growth, while relatively smooth when tilted toward $\langle 11\bar{2}0 \rangle$.^{7,8} This may be because of instability of steps perpendicular to the $\langle 11\bar{2}0 \rangle$ direction.⁹ With a larger off-angle, however, a group reported that such undulations were not observed.¹⁰ Now we assume that the etching process be inverse step-flow growth. Atomic bonds near the $\langle 01\bar{1}0 \rangle$ -off surface should be as shown in Fig. 4: each of the three adjacent Si–C bilayers has two dangling bonds at the step edge, and each of the next three bilayers has only one dangling bond. Although it is not clear which three bilayers are etched faster, either three bilayers should be etched faster as shown in Fig. 5. As a result, “fast-etched” steps catch up to the adjacent “slow-etched” steps, forming microsteps with six-bilayer height. The formation of the six-bilayer-height microsteps may be realized easily when the off direction is $\langle 01\bar{1}0 \rangle$ as shown in Fig. 5(a). If the off direction is except for $\langle 01\bar{1}0 \rangle$, and in usual circumstances such as H_2 etching,⁴ the steps perpendicular to $\langle 01\bar{1}0 \rangle$ family should have the lowest surface energy among all the directions in the (0001) basal plane, i.e., be etched at the lowest rate, leaving steps perpendicular to $\langle 01\bar{1}0 \rangle$ family. Thus, except for the off direction toward $\langle 01\bar{1}0 \rangle$, zigzag steps perpendicular to $\langle 01\bar{1}0 \rangle$ family should be formed as shown in Fig. 5(b). In the case of 6H–SiC, there are two

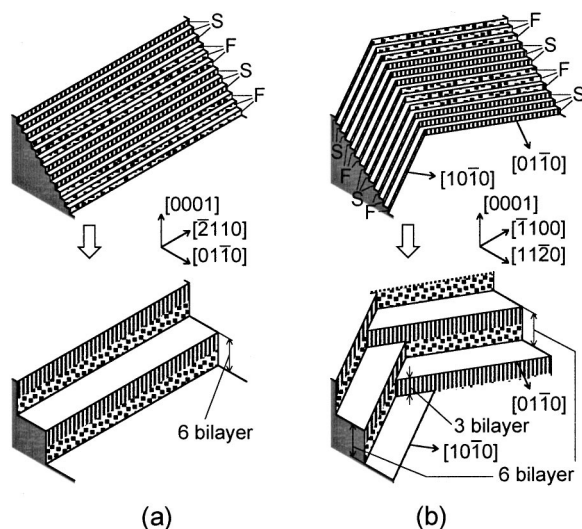


FIG. 5. Shapes of the steps on (a) $\langle 01\bar{1}0 \rangle$ -off substrate and (b) $\langle 11\bar{2}0 \rangle$ -off substrate. The letters F and S indicate fast- and slow-etched steps, respectively.

kinds of steps with different etch rate mentioned above, and the etch rate changes every third bilayer. As a result, zigzag microsteps are formed, but the terraces between the steps perpendicular to $[01\bar{1}0]$ and the terraces between the steps perpendicular to $[10\bar{1}0]$ have three-bilayer offset in height: that is, interlacing. In the case of HCl etching, however, straight steps were observed even on $\langle 11\bar{2}0 \rangle$ -off substrates as shown in Fig. 2. Hence, the etch rate toward $\langle 11\bar{2}0 \rangle$ should

be as slow as, or even slower than, toward $\langle 01\bar{1}0 \rangle$ under some circumstances. The reason of this difference in the etch rate is not clear yet, but probably due to the presence of Cl adatoms. As another feature, alternation of two- and one-bilayer-height steps were observed on some $\langle 11\bar{2}0 \rangle$ -off substrates as shown in Fig. 3(b). The cause may be attributed to the difference of bond configuration, i.e., hexagonal and cubic site.

In summary, step bunching on 6H-SiC (0001)-vicinal face etched by HCl was investigated by AFM. When the substrate had the inclination toward near $\langle 01\bar{1}0 \rangle$ or even $\langle 11\bar{2}0 \rangle$, continuous parallel and periodic microsteps with six-bilayer height were laid perpendicular to the off direction, although with the inclination toward $\langle 11\bar{2}0 \rangle$, the microsteps were apt to decompose into three-bilayer or less and inhomogeneity of step structure was observed in different samples or even in one sample.

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